NIST Develops a Transition Edge Sensor Microcalorimeter for Quantitative Analysis

NIST researchers have invented and developed the Transition Edge Sensor (TES) Microcalorimeter, a relatively new method of detection that is capable of high resolution over a broad energy range. NIST is working with a small US company who is manufacturing a commercial version of the invention. There has already been considerable interest and inquiry into the availability of the detector. In addition, at NASA's request, NIST is participating in a study on the feasibility of TES Microcalorimeter incorporation into the lunar exploration program.

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Quantitative analysis is at the heart of chemistry and materials science. On the microscopic scale, quantitative analysis with an electron microscope or microbeam probe has become the standard technique in universities and industry. It has, for example, played a key role in the development of our electronics industry. The standard techniques for analysis of characteristic x-rays produced by the beam of an electron microscope are the energy dispersive (ED) methods (represented by the lithium-drifted silicon detector and silicon drift detectors) and the wavelength dispersive (WD) methods (detectors based on x-ray diffraction). ED methods cover a wide energy range but have poor resolution, while WD methods have high resolution over a narrow energy range.

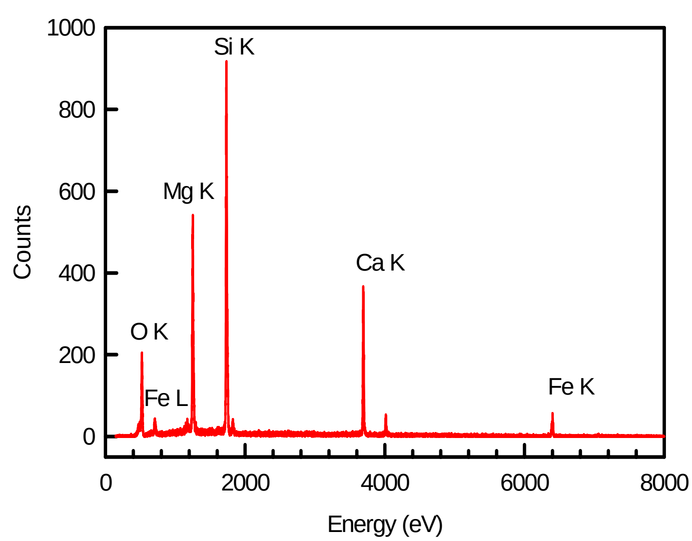
NIST's (TES) Microcalorimeter incorporates a small x-ray absorber cooled to very low temperatures (<100 mK) and a thermometer capable of measuring the temperature rise from the absorption of single x-ray photons. The thermometer, a thin film of metal maintained at its superconducting transition, is capable of a remarkably linear response that makes it ideal for the energy resolution of characteristic x-rays from nearly all chemical elements. It was initially subject to the kind of problems typical of a new and sensitive measurement technology, we have overcome the most troublesome issues and have proceeded this year to prove the detector's value in scientific applications. To goal of the work described here was to demonstrate the ability of the detector to conduct quantitative analysis in situations that would have been previously impossible.

The microanalysis of elements with an electron probe is a primary goal of the TES Microcalorimeter program. The microcalorimeter detector has been incorporated into a dedicated JEOL 840 electron microprobe for this purpose. This year we conducted several sets of measurements on NIST SRM glasses (K411 and K458) to compare the accuracy of Microcalorimeter measurements with the chemical assays which certify these SRM's.

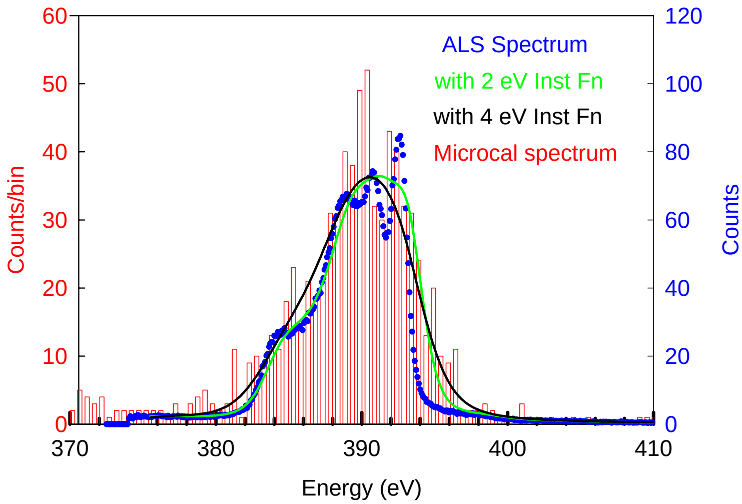
The accuracy of the TES Microcalorimeter was verified using NIST Standard Reference Materials providing reference values. The high resolution capability was verified when x-ray emission from a chemical compound was observed with sufficient resolution to identify its valence band.

In this work, we quantitatively determined the concentrations of Ba, Ca, Fe, Mg, O, Si, and Zn over an energy range of 500 eV—8000 eV. Characteristic x-ray line intensities of K and L transitions were measured in spectra from standard compounds and from the materials to be analyzed. In both, we included lines from the above elements which lie so close together in energy as to interfere with each other. The excellent energy resolution, combined with the broad energy range, allowed us to carry out of element concentration determinations. These determinations would be unattainable by single spectra from conventional detectors. The overall discrepancies—1% in total concentration, and a maximum of 8% in relative concentration of a weak element—are quite acceptable for many analyses.

The figure illustrates a spectrum obtained in 1000 s of live counting time.



We also obtained an x-ray fluorescence spectrum from a compound which demonstrates chemical specificity. As a test case, we selected the chemical compound BN in which the excited state of a N 1s core hole is directly filled with valence electrons—2s and 2p electrons from the N atom, as well as with valence electrons from other atoms in the compound. The hybridization of these atomic states into molecular orbitals results in a valence electron density of states that extends over approximately 30 eV and which is specific to this compound. The resulting emission spectrum extends over a similar energy width. We compared the x-ray emission spectrum by the Microcalorimeter with a high-resolution spectrum that we obtained on a grating spectrometer beamline at the Advanced Light Source at Lawrence Berkeley Laboratory. A comparison between the TES Microcalorimeter spectrum and the light source spectrum broadened to the same 4 eV resolution confirmed that we are obtaining accurate evidence of the valence band.



The figure to the left shows the valence band x-ray emission spectrum of BN from the microcalorimeter and from beamline 8.0.1 of the Advanced Light Source, broadened to 2 eV and 4 eV resolution.

Future Plans: This activity has a broad future ahead of it. The program of quantitative analysis will continue to determine the limits of its applicability during the current fiscal year. The program to obtain high resolution valence band x-ray emission spectra for chemical analysis will be augmented by the design and construction of a new detector which will cover an energy range of about 1 keV with 1 eV resolution.

Selected for Advanced Publication:

T. Jach, N. Ritchie, J. Ullom, and J. A. Beall, **"Quantitative Analysis with the Transition Edge Sensor Microcalorimeter X-ray Detector,"** *Advances in X-ray Analysis, 50,* 2007